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**2 modified binder at reduced temperature**

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# Porous asphalt mixture with alternative aggregates and crumb-rubber modified binder at reduced temperature

## Abstract:

This paper studies the design and characterization of a PA mixture with 91% of EAF slag, using a commercial CRM binder. A fatty acid amide wax was added to decrease the mixture manufacturing temperature. The mechanical performance of the designed mixtures was studied with the determination of void characteristics, water sensitivity, and particle loss in dry and wet conditions. Finally, their compactability, stiffness and fatigue resistance were also analysed.

The addition of the wax allowed to decrease the manufacturing temperature 15 °C. Besides, the wax increased the complex modulus of bitumen, increasing also the elastic component and decreasing the thermal susceptibility, although these modifications did not have a significant impact in the mechanical performance of the mixture.

**Keywords:** porous asphalt mixture; crumb-rubber modified bitumen; wax; EAF slag; warm mix asphalt.

## Highlights:

- PA mixtures with 91 % of alternative aggregate have been designed
- The influence of a fatty acid amide was on the CRM binder was analysed
- The wax decreases 15 °C the manufacturing temperature of the asphalt mixture
- The wax increases stiffness and decreases bitumen thermal susceptibility
- Similar mechanical performance has been observed for PA with and without the wax

## 1. Introduction

Road infrastructure is a resource-intensive sector since a large amount of materials and energy are required during the construction, maintenance and rehabilitation of pavements. Hence, the search of cost-effective and eco-friendly practices will result in a huge impact. The development of new techniques and the use of alternative materials can significantly contribute to decrease the environmental impact of asphalt mixes. As an example, warm mix asphalts (WMA), manufactured by different methods [1,2], have

revolutionized the construction process of the road sector by means of reducing the air emissions at both the asphalt plant and the construction site, thus decreasing the environmental impact and improving the working conditions. Likewise, replacing the natural aggregate with alternative materials is another strategy widely used. Thus, recycled asphalt pavement (RAP) from roads[3], by-products[4,5] or construction waste [6] are some of the materials used in bituminous mixtures to replace natural aggregate, although other are also standing out, as rubber from end-of-life tires[7,8] and other polymers[9,10], which can also be used to improve the mechanical performance of the bituminous mixtures.

Therefore, although the impact of all these strategies have been analysed in different studies, the combined use of these practices to improve the environmental impact of asphalt mixes has not been sufficiently studied together.

This paper addresses the design of a porous asphalt (PA) mixture at reduced temperature and incorporating high percentage of recycled aggregates and crumb-rubber modified (CRM) bitumen. Electric Arc Furnace (EAF) slag aggregate was selected to replace most of the natural aggregate[11], because this material shows a great resistance against fragmentation and polishing[12]. A commercial crumb-rubber modified (CRM) bitumen was used as binder. The Spanish normative prioritizes this type of binder because the incorporation of crumb rubber from end-of-life tyres contributes to the Spanish resource-efficiency policy. On the other hand, the addition of rubber improves the properties of the bitumen, increasing the elasticity and decreasing the thermal sensitivity[13]. However, the higher viscosity of this bitumen force the mixture to be produced at a greater temperature, which increases the energy consumption and the greenhouse gas emissions to air [14], hindering its use. Therefore, a fatty acid amide wax was used to neutralize this effect. Although the impact of organic additives in this type of bitumen is smaller than the one observed in conventional binders [15], the aim in this study was to achieve the same manufacturing conditions of conventional 50/70 penetration grade bitumen.

For the development of this research, which involves the elements previously described, a porous asphalt mixture type was selected. Its particle size distribution fits perfectly with the EAF slag aggregate, which usually contains a low percentage of fines. Besides,

the porous asphalt mixtures also show some environmental advantages, as a better management of surface run-off[16] and a significant decrease of road noise[17].

The aim of this paper is to demonstrate the technical viability of a porous asphalt mixture with a high percentage of alternative aggregates, and manufactured with a CRM binder but at reduced temperature, trying to achieve the same manufacturing conditions than a conventional 50/70 penetration grade binder.

## 2. Materials and methods

### 2.1 Materials characterization

A previous analysis was carried out to find out the properties of the materials. An EAF slag from a steel factory of Cantabria (Spain) was used as coarse aggregate. Table 1 shows its main characteristics and the limits of the Spanish standard for the highest heavy traffic category.

Table 1. EAF Slag properties

Property	Result	Spanish standard	Specification
Specific weight ( $\text{g}/\text{cm}^3$ )	3.821	-	EN 1097 – 6
Water absorption WA 24 (%)	1	-	EN 1097 – 6
Slab index	2	< 20	EN 933 – 3
Los Angeles coefficient	18	$\leq 20$	EN 1097 – 2
Polished Stone Value	0.59	$\geq 0.56$	EN 1097 - 8

According to the results, the material showed good properties as coarse aggregate. The low coefficient of Los Angeles guarantees a hard mineral skeleton and the high PSV value means a superior skid resistance of the road surface, what is an important safety road factor. Besides, the potential expansiveness (EN 1744-1) and leaching of contaminants (EN 12457-4) were analysed and the material and complied with current normative for their use in asphalt mixes in Spain [18,19]. As expected, the EAF slag aggregates presented higher specific weight than conventional aggregates. Regarding the fine fraction, it was completed with limestone, with a density of  $2.708 \text{ g}/\text{cm}^3$ , and the sand equivalent coefficient was 78.

The bitumen was a PMB 45/80 – 60C, which has approximately 10 % of rubber, and according to the supplier, a manufacturing temperature between 165 °C and 175 °C. Its main properties are presented in the Table 2.

*Table 2. Characteristics of CRM binder*

Property	Result
Penetration (0.1 mm)	54
Softening point (°C)	63
Elastic recuperation (%)	58
Relative density (g/cm <sup>3</sup> )	1.047

Finally, a fatty acid amide wax (Kemfluid) was selected to decrease the manufacturing temperature. This additive is manufactured in Zaragoza (Spain) from pig tallow and presents a melting point around 140 °C[20].

## **2.2 Viscosity and DSR test**

The wax was used to decrease the viscosity of the binder. Its impact on the bitumen was studied with a rheometer DHR-1 of TA Instrument, which was used to analyse the rheological behaviour of the bitumen with and without the wax. Both tests were carried out with a 25 mm plate geometry and a 1 mm gap. The viscosity test was performed with a temperature ramp from 100 °C to 190 °C in rotational mode, while the DSR test was done in oscillatory mode from 0.1 Hz to 10 Hz with a 0.1% strain, in a range of temperatures from 20 °C to 75 °C at 5 °C intervals.

In all the samples with wax, the percentage added was always 3 % of bitumen weight, while the mixing process was carried out at 150 °C with an IKA homogenizer during 5 minutes at 15,000 rpm.

The decrease in the production temperature that it is possible to achieve by adding the wax was determined by measuring the dynamic viscosity between 100 °C and 190 °C. The viscosity of the CRM binder without wax at the manufacturing temperature recommended by the supplier (170 °C) was considered as reference. The test was repeated under the same conditions to the samples with wax. The temperature, at which the samples achieved the reference viscosity, was considered the new reduced manufacturing temperature. Besides, the master curves of binder stiffness and phase

angle were obtained to analyse the influence of the wax on the performance of the CRM binder, so the rheological behaviour of both bitumen was analysed independently from the frequencies and temperatures used in the test[21,22].

### **2.3 Design of PA mixture**

Firstly, porous asphalt mixes incorporating EAF slag aggregates as coarse aggregate and CRM bitumen were designed according to the Spanish standards, at the conventional temperature recommended by the bitumen provider. In order to ensure that the mixes comply with the mechanical requirements, the following tests were carried out: The void characteristics of bituminous specimens (EN 12697 – 8), water sensitivity test (EN 12697 – 12) and Cantabro loss particle test in dry (EN 12697 – 17) and wet conditions (NLT-362 Spanish Standard). As a second step, the fatty acid amide wax was added to the same PA mix composition and new samples were produced at the reduced temperature previously determined with the rheometer. The same mechanical tests were repeated to the samples to assess the potential effect of reducing the production temperature in their mechanical performance.

The requirements set by the Spanish regulations for the highest traffic level were considered as the reference for the mechanical performance. On the other hand, dynamic tests were also done to better characterize the performance of the mixtures. Thus, the asphalt mixes were tested for stiffness (EN 12697 – 26), fatigue resistance (EN 12697 – 24), and energy compaction (EN 12697 – 31). By this way, the impact of the wax on the rheological performance of the bitumen can be related with the impact on the mechanical performance of the asphalt mixture.

### **2.4 Statistical analysis**

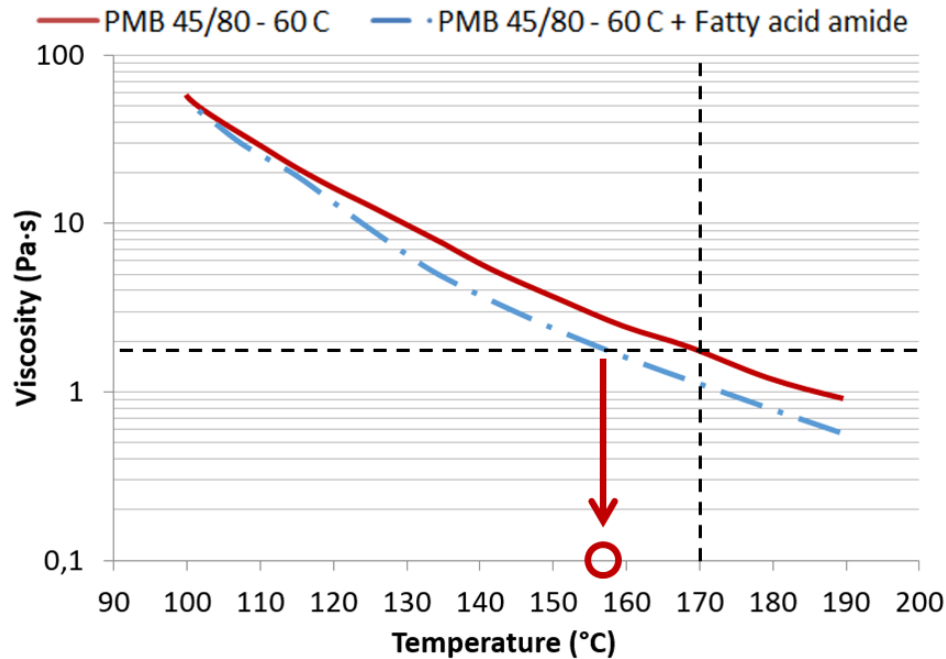
The Minitab software was used to determine the statistical significance of the reduction in the production temperature. The confidence interval considered was 95 % (p-value of 0.05). In those cases, where the results fulfilled a normal distribution and there was homogeneity of variances the T Student test was applied. Otherwise, the U of Mann-Whitney test was used.

### 3. Results and Discussion

#### 3.1 Rheological analysis

The viscosity test (Figure 1) was carried out with three samples of each bitumen to determine the reduction of the asphalt production temperature that it is possible to achieve by adding the wax.

Figure 1. Result of the viscosity test



As it can be observed in Figure 1, the addition of the fatty acid amide wax decreases the viscosity of the CRM bitumen when the temperature of the mixture is above the melting point of the wax (140 °C). However, when the temperature falls below 130 °C the viscosity of the CRM bitumen with wax starts rising, reaching the viscosity of the original CRM bitumen at around 100 °C. Therefore, the behaviour of the reference bitumen with the wax can be divided in two zones:

Zone A. Above the melting point of the wax (from 140 °C to 190 °C), where the viscosity of the bitumen with wax is below and parallel to the reference bitumen.

Zone B. Under the melting point of the wax (from 100 °C to 140 °C), where the viscosity of the bitumen with the wax increases faster than the viscosity of the reference bitumen.

The curves of viscosity obtained were adjusted to Arrhenius equation, where  $\mu$  (Pa·s) is

the viscosity,  $T$  is the temperature in kelvin degrees,  $E_f$  (J/mol) is the flow activation energy,  $R$  is the universal gas constant ( $8.314 \text{ J/mol} \cdot \text{K}$ ), and  $A$  is the fitting parameter[1].

$$\eta = A \cdot e^{\frac{E_f}{R \cdot T}} \quad (1)$$

Table 3 presents the Arrhenius equation for the reference bitumen with and without the wax in the two different zones: above (A) and under (B) the wax melting temperature.

*Table 3. Viscosity curves and activation energy for both bitumen and zones*

Binder	Zone	Equation	$E_f$ (J/mol)	$R^2$
PMB 45/80-60 C	A.	$\eta = 153.464 \cdot 10^{-9} \cdot e^{\frac{7188.73}{T}}$	59767	0.99
PMB 45/80-60 C + Fatty acid wax		$\eta = 168.539 \cdot 10^{-9} \cdot e^{\frac{6948.74}{T}}$	57771	0.99
PMB 45/80-60 C	B.	$\eta = 10.231 \cdot 10^{-9} \cdot e^{\frac{8317.61}{T}}$	69152	0.99
PMB 45/80-60 C + Fatty acid wax		$\eta = 0.058 \cdot 10^{-9} \cdot e^{\frac{10266.62}{T}}$	85356	0.99

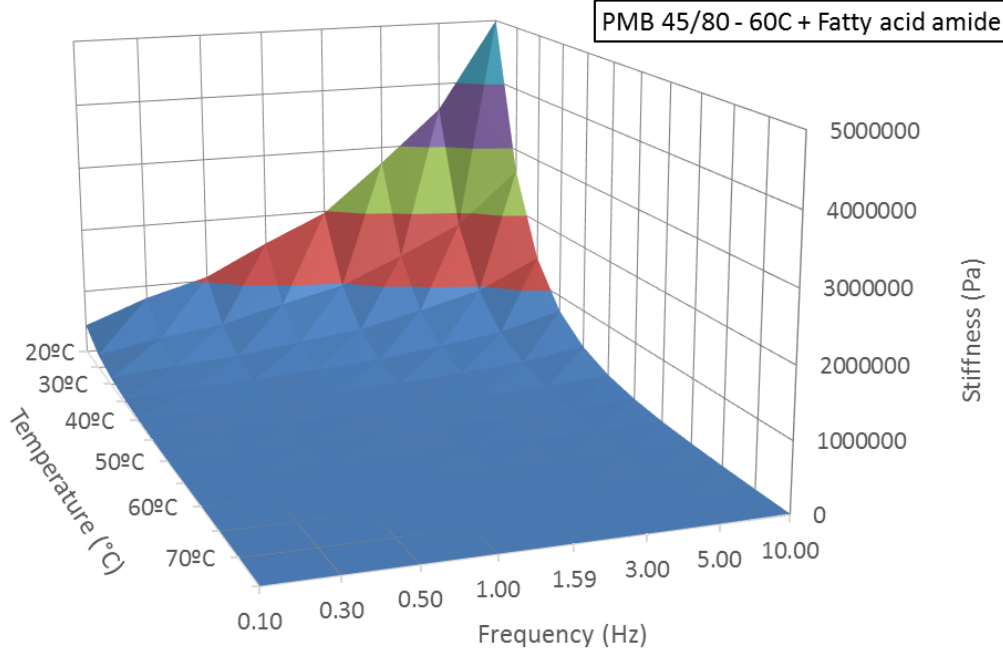
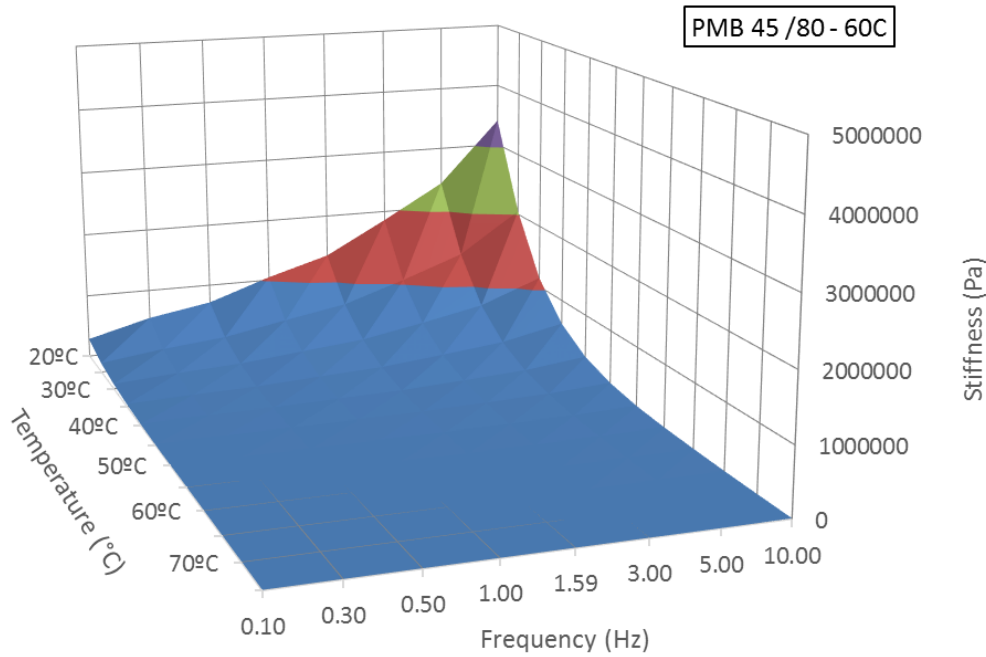
These curves were used to calculate the temperature at which the bitumen with wax reaches the same viscosity than the original CRM bitumen at its recommended production temperature ( $170^\circ \text{C}$ ). According to the results, the temperature could be reduced by  $15^\circ \text{C}$ ; so, the asphalt mixture with wax was manufactured at  $155^\circ \text{C}$ .

In zone A, in the case of the bitumen with wax, a slight decrease of the activation energy is observed, meaning that less energy is required for molecular movement when the temperature is higher than the melting point of the wax. On the other hand, when the temperature is below the melting point of the wax (zone B), and the wax change from liquid to solid, the resistance to flow of the bitumen/wax mix increases. This is clearly reflected in the change of slope that is produced between  $130^\circ \text{C}$  and  $140^\circ \text{C}$ .

As described before, the rheological behaviour of both bitumen was analysed to evaluate the influence of the wax on the performance of the CRM bitumen. The stiffness ( $G^*$ ) and phase angle ( $\delta$ ) of the reference bitumen PMB 45/80 – 60C and this bitumen with 3 % of fatty acid amide wax are shown in Figure 2 and Figure 3.

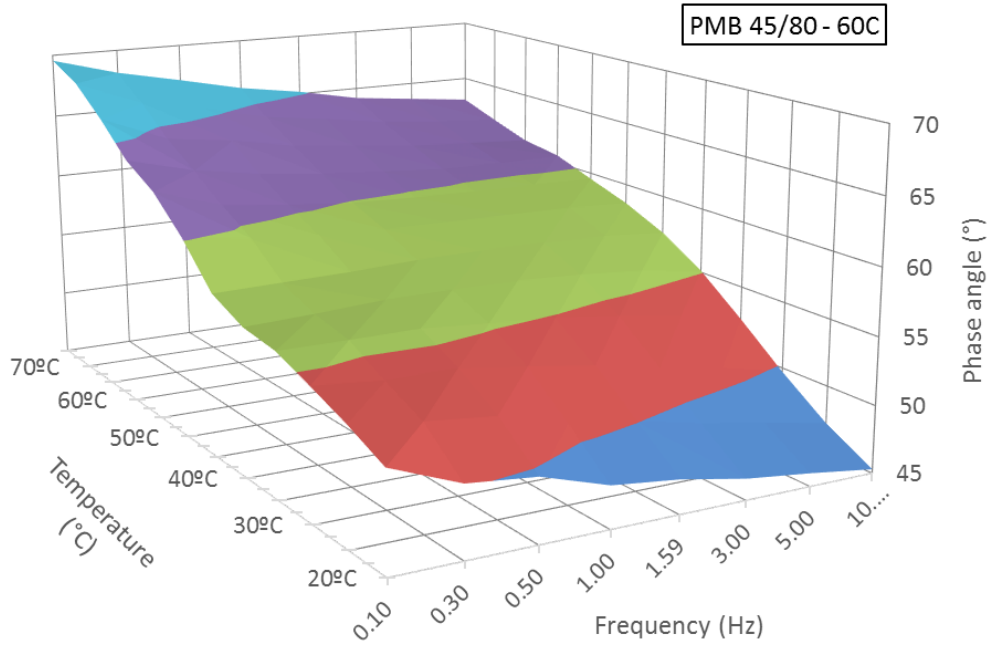
*Figure 2. Stiffness. From up to down: reference bitumen and reference + Fatty acid amide*



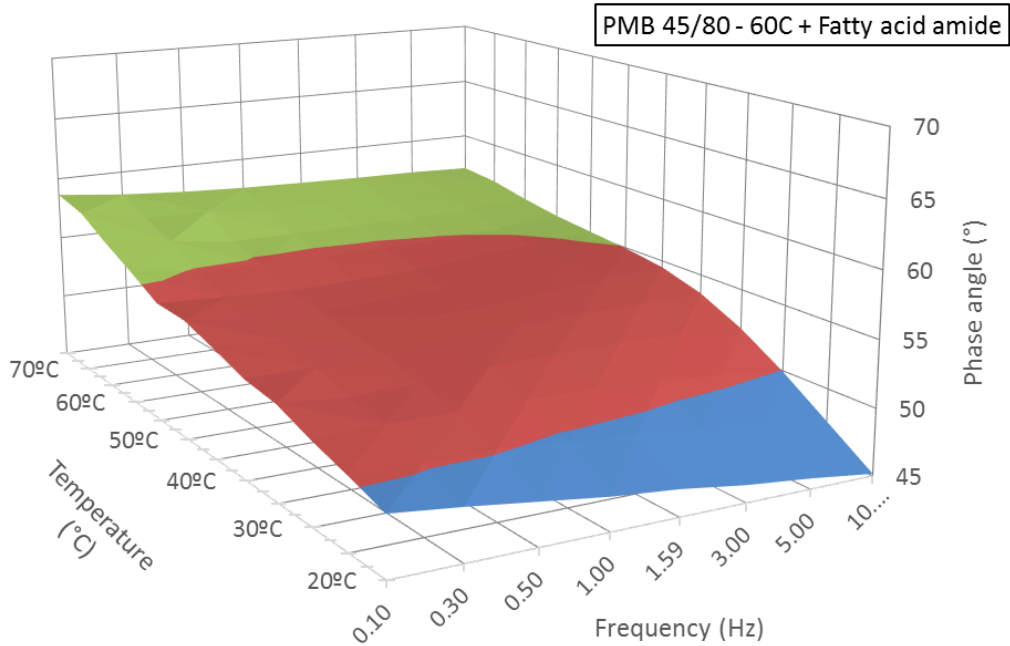


As can be observed, the addition of the wax increased the stiffness, especially at low temperatures and high frequencies, and decreased the phase angle, suggesting a more elastic behaviour of the bitumen with wax. However, unlike in the case of the stiffness, the greater differences in the phase angle are produced at high temperatures and low frequencies. On the other hand, the relation of the phase angle with the temperature and frequency also changed, since a more horizontal plane is obtained, which implies that the bitumen with the wax is less dependent of these parameters.

Figure 3. Phase angle. From up to down: reference bitumen and reference + Fatty acid amide



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199 Likewise, the master curve for both bitumen was developed. The stiffness results were  
200 adjusted to a sigmoidal curve by least-squares fitting:

$$\text{Log } G^*(\text{Pa}) = \alpha + \beta / (1 + \exp(\rho - \gamma \cdot \log \omega_r)) \quad (2)$$

201 Where  $\alpha$  is the lower asymptote,  $\beta$  is the difference between the values of upper and  
202 lower asymptote,  $\rho$  and  $\gamma$  are shape parameters (they define the position of the turning  
203 point and the slope respectively)[23] and  $\omega_r$  is the reduced frequency:

$$\omega_r = a_T \cdot \omega \text{ (rad/s)} \quad (3)$$

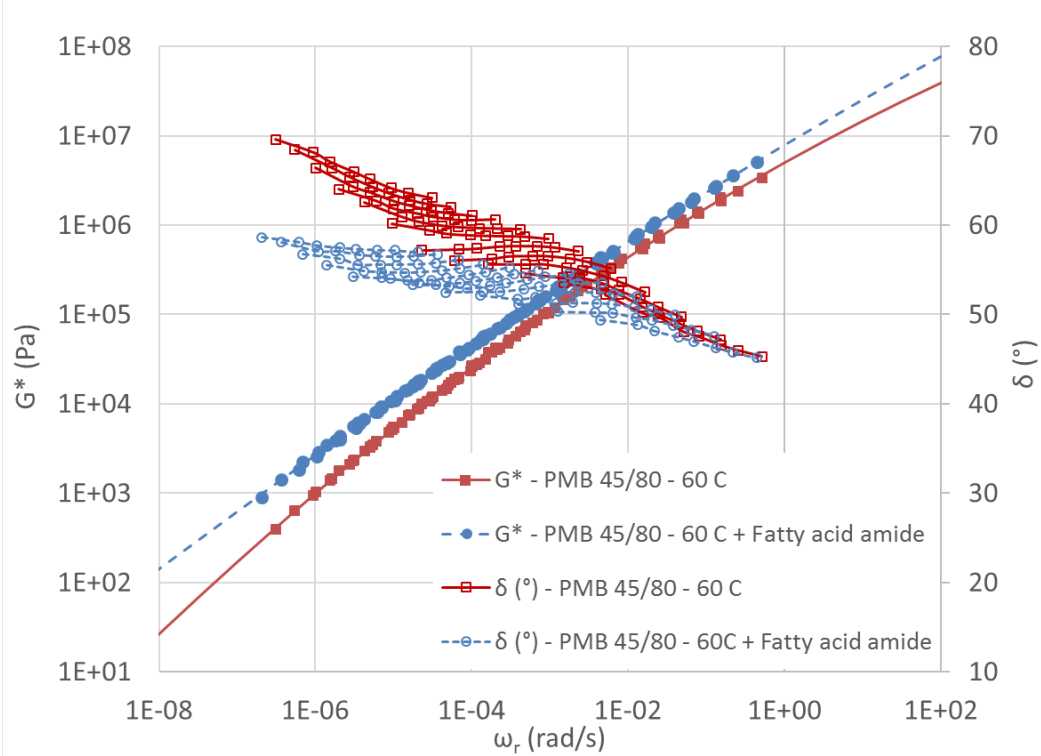
204  $\omega_r$  links the frequencies of the test ( $\omega$ ) with the temperature ( $a_T$ ):

$$a_T = a_1 \cdot T(^{\circ}\text{C})^2 + a_2 \cdot T(^{\circ}\text{C}) + a_3 \quad (4)$$

205 Where  $a_1$ ,  $a_2$  and  $a_3$  are shape parameters.

206 The same shift factors derived for the stiffness were used to obtain the master curve of  
207 the phase angle. These master curves are shown in Figure 4.

208 *Figure 4. Master curves of CRM bitumen alone and with the fatty acid amide wax*



209 According to this figure, a higher complex modulus is obtained for all the reduced  
210 frequencies when the wax is added to the CRM bitumen, which is in agreement with the  
211 work of other authors[2,24]. The greatest difference in the complex modulus of the  
212 reference bitumen and the bitumen/wax mixture is produced at low reduced  
213 frequencies (or high temperatures), so this increase of the binder stiffness should  
214 improve the resistance against permanent deformation. However, when adding the  
215 wax, a slight increase of the stiffness is also observed at high reduced frequencies (or  
216 low temperatures), what could imply that the bitumen is more prone to cracking.

218 On the other hand, the wax decreases the phase angle associated to each modulus,  
219 making the binder more elastic especially at low reduced frequencies, as it was  
220 previously explained in Figure 3. It should also be noted that both binders show a lack

of linearity probably due to the modification of the bitumen structure caused by the rubber. The CRM bitumen shows a slight “S” shape traditional of this type of bitumen, when the wax is added this shape disappears, probably due to the interactions between it and the polar fractions of bitumen (asphaltenes and resins)[24].

The incorporation of the wax increases the solid-like behaviour, as stated by other studies[14,24].

The parameters of the master curve and the correlation coefficients are shown in Table 4. In both cases, the value of  $\alpha$  is negative, which means that  $G^*$  at low frequencies (or high temperatures) is very small. The asymptotes of the bitumen with wax are upper than asymptotes of reference bitumen, which coincides with its increase of the stiffness.

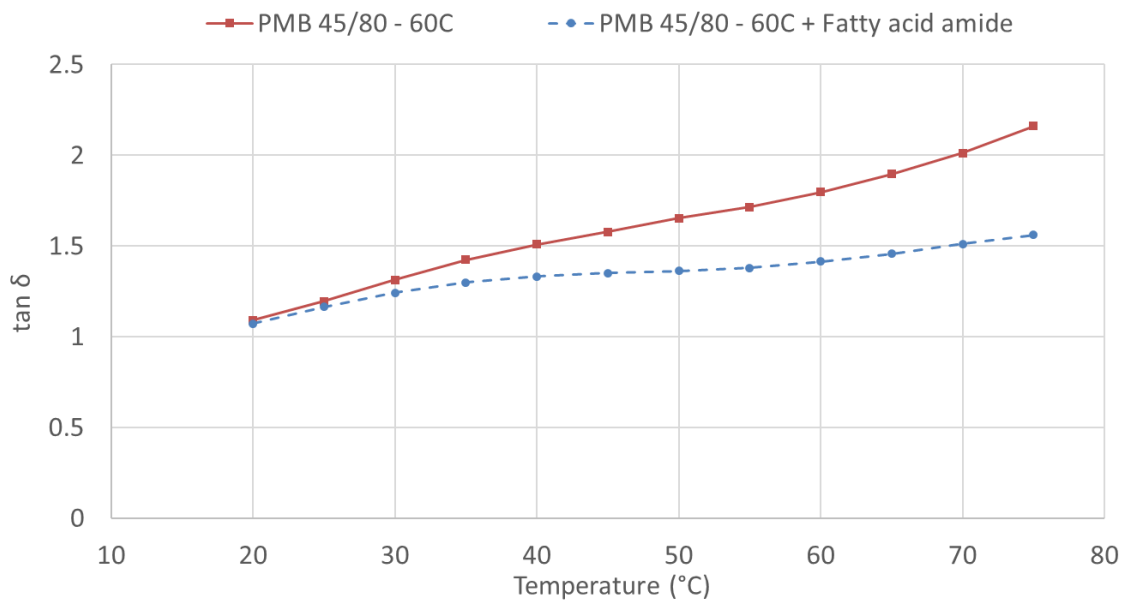
*Table 4. Master curve parameters*

	$\alpha$	$\beta$	$\rho$	$\gamma$	$a_1$	$a_2$	$a_3$	$R^2$
PMB 45/80 – 60C	-18.85	30.21	-1.70	0.12	$5.72 \cdot 10^{-4}$	-0.13	0.30	0.997
PMB 45/80 – 60C + fatty acid amide	-12.60	26.87	-0.97	0.10	$5.80 \cdot 10^{-4}$	-0.13	-0.30	0.998

The increase in the  $\rho$  parameter when the wax was added means that the horizontal position of its turning point increased. However, this did not imply a reduction of the hardness of the bitumen with wax as it was stated by other author[25]), because the master curve of the bitumen with wax is always above the reference bitumen due to the differences in the position of the asymptotes. The slope is quite similar in both cases.

Finally, the thermal susceptibility was also analysed with the value of  $\tan(\delta)$  [26]. A flat curve implies a lower susceptibility of temperature. The values of  $\tan(\delta)$  are presented in Figure 5 for both bitumen at 1.59 Hz, which has been considered as the representative frequency. According to the results, the thermal susceptibility is lower when the wax is added, which is linked with the lower phase angle obtained in the rheology analysis.

*Figure 5.  $\tan(\delta)$  values of both bitumen*



The rheological analysis of the bitumen samples showed that the incorporation of fatty acid amide wax increases the modulus and the elastic behaviour of CRM bitumen, makes the binder less sensitive to thermal variations, and decreases the manufacturing temperature approximately by 15 °C.

### 3.2 Mechanical tests

Taking into account the high specific weight of the EAF slag aggregate and in order to design well-balanced mixes, the design of the mixture was carried out by volume. Therefore, although due to the high specific weight of the EAF slags, the density of the resulting mixes is higher than the density of conventional PA mixes (above 2.5 g/cm<sup>3</sup>) and the percentage of bitumen by weight is lower than usual, the final quantity of bitumen is in the range of conventional porous asphalt mixtures.

Concerning the particles size distribution, no slag aggregate was used below 2 mm sieve, only limestone. The particle size distribution of the PA mixes is presented in the Figure 6 and the percentage of each material is shown in the Table 5. The percentage of bitumen was 3.85 % by weight of mixture; what it is approximately 50 g of bitumen per Marshall sample.

Figure 6. Particle size distribution of PA mixture

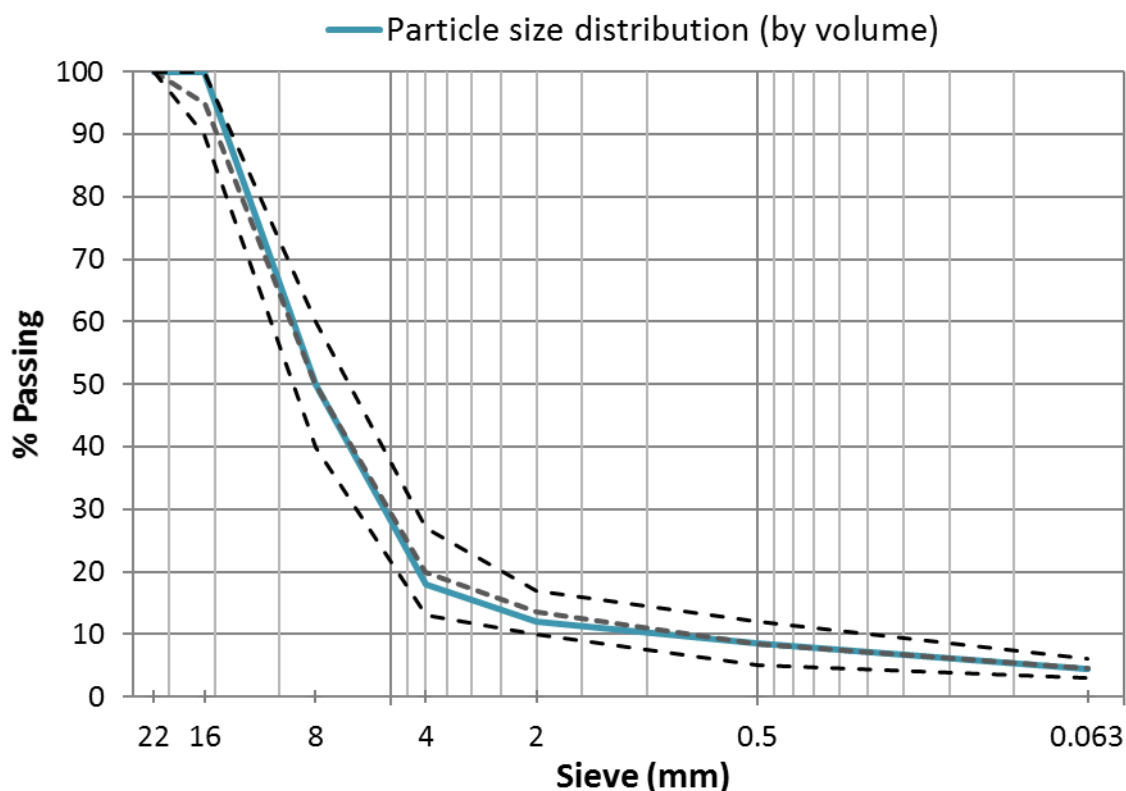


Table 5. Percentage of each material

Material	% by volume	% by weight
Slag	88	90.9
Limestone	7.5	5.5
Limestone Filler	4.5	3.6

As previously explained, the porous asphalt mixture incorporating the original CRM bitumen was manufactured at 170 °C, as recommended by the supplier. On the other hand, the PA mixture containing the CRM bitumen/wax mix was produced at 155 °C. Both PA mixes were subjected to the same mechanical; therefore, there are two equivalent mixtures: one manufactured at 170 °C (conventional temperature), and another with the wax manufactured at 155 °C (reduced temperature).

#### *Determination of void characteristics of bituminous specimens (EN 12697 – 8)*

The void characteristics of the mixtures is presented in Table 6. The mixture produced at reduced temperature presented a slightly higher air void content, despite the viscosity should be the same in both mixes. On the other hand, the statistical analysis indicated that this increment was not significant (Table 9).

274

Table 6. Void characteristics of both mixtures

Temperature	170 °C	155 °C	Spanish Standard
Density (g/cm <sup>3</sup> )	2.608	2.547	-
Voids in mixture (%)	22.2	24.4	≥ 20

275 *Water sensitivity test (EN 12697 – 12)*

276 No mixture was affected by the water saturation, since both mixtures achieved a high  
 277 Indirect Tensile Strength Ratio (ITSR). The results are presented in Table 7. Although the  
 278 mixture manufactured at reduced temperature had a good behaviour with a slightly  
 279 higher ITSR, this mixture reached lower Indirect Tensile Strength (ITS). This decrease in  
 280 the resistance agrees with other studies [27]. The ITS of the dry samples was significantly  
 281 lower in the case of the mixtures with wax (p-values are shown in Table 9), although this  
 282 parameter could have been affected by the difference in the percentage of voids.  
 283 However, in the case of the wet samples, there were not significant differences among  
 284 the mixtures, so it seems that the water saturation affects, at least equally, to both  
 285 mixtures.

286

Table 7. Water sensitivity test

Temperature	170 °C	155 °C	Spanish Standard
I.T.S. (KPa)	Dry 886.0	764.0	-
	Wet 805.2	706.2	-
I.T.S.R. (%)	91	92	≥ 85

287 *Cantabro loss particle test in dry (EN 12697 – 17) and wet conditions (NLT-362)*

288 This test was carried out under two different conditions: dry samples were used for the  
 289 determination of the resistance against abrasion, while the loss of cohesion caused by  
 290 water was evaluated in wet conditions. The results for both temperatures were very  
 291 similar (Table 8), although a slightly increasing trend in the particle loss is observed when  
 292 the mixture is produced at reduced temperature. This can also be attributed to the small  
 293 difference of voids, increasing the percentage of mass loss proportionally to the  
 294 percentage of voids. In any case, the resulting differences were not statistically  
 295 significant (Table 9).

296

Table 8. Cantabro particle loss test

Temperature	170 °C	155 °C	Spanish Standard
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Dry samples (%)	12.8	14.4	≤ 20
Wet samples* (%)	29.6	33.5	≤ 35

\*Required until 2008.

According to the results obtained in these mechanical tests, the two PA mixtures fulfilled the requirements established in the Spanish regulations for the most demanding conditions (highest traffic level and warmest area). From the mechanical point view, the EAF slag presents an excellent performance as coarse aggregate, and the use of a fatty acid amide wax to reduce the production temperature does not significantly modify the mechanical properties of the mixture, given that the performance of the porous asphalt mixture at conventional and reduced temperature was statistically equivalent.

The results followed a normal distribution and there was homogeneity of variances except in the case of the Cantabro particle loss test in wet conditions. The T of Student and U of Mann-Whitney tests were applied respectively. In Table 9 the p-values of each test are shown. The ITS of the dry samples in the water sensitivity test is the only statistically different result, with a p-value under 0.05, although the p-value of the void characteristics is also close to the 0.05 limit.

*Table 9. Significances of mechanical test of porous asphalt mixture at conventional and reduced temperature*

	Voids	Water sensitivity		Loss particle	
		dry	wet	dry	wet
P-value	0.052	0.013	0.066	0.535	0.665

### **3.3 Compactability**

The compactability test (EN 12697-10) was carried out to analyse if a higher level of compaction energy is required when the mixture is manufactured with the wax at 155 °C. The test was performed with a Controls ICT 76-B0251 gyratory machine on three samples of 100 mm of diameter per type of mixture, the load was 600 KPa, the speed of movement 30 rpm and the angle of rotation 0.82°. The accumulated energy was calculated using the model developed by *del Rio*[28]:

$$\frac{W}{m} = \sum_{i=1}^N \frac{W_i}{m} = \frac{2 \cdot \pi \cdot \alpha \cdot A}{m} \sum_{i=1}^N h_i \cdot S_i \quad (5)$$

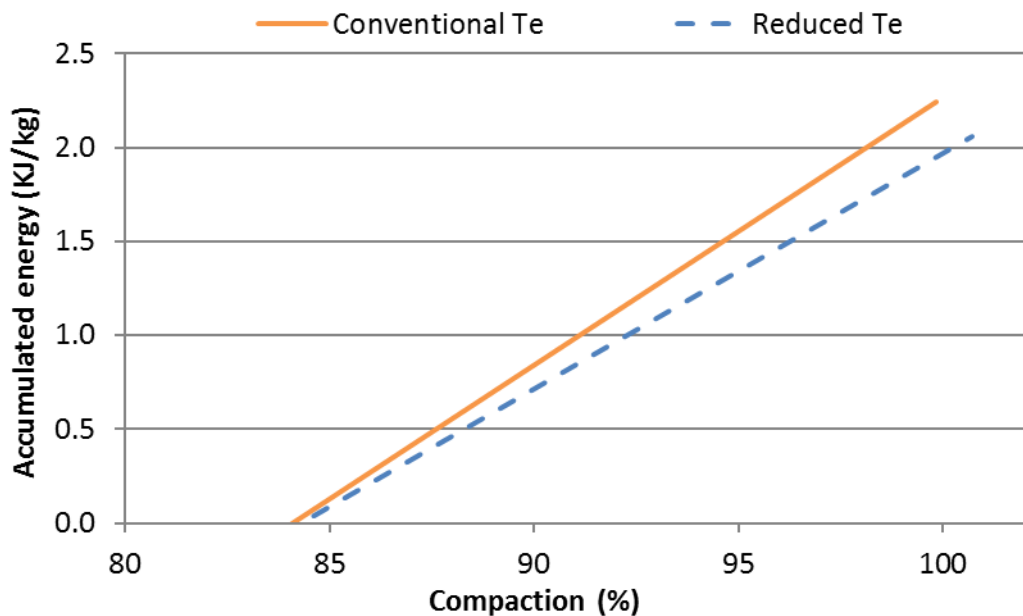


Where:

- $W$  (KJ): energy of compaction;
- $m$  (Kg): mass;
- $N$ : total cycles applied;
- $\alpha$  (rad): inclination angle of the cylindrical sample;
- $A$  (m<sup>2</sup>): Transverse area of the sample;
- $h_i$  (m): height of the sample in each cycle  $i$ ;
- $S_i$  (KN/m<sup>2</sup>): shear stress measured in each cycle  $i$ ;

The required compaction energy is shown in Figure 7:

Figure 7. Required compaction energy for each type of mixture



A slightly lower energy is needed for compacting the PA mixture at 150 °C. The results were adjusted using the linear least-squares method (Equation 6), whose characteristics are shown in Table 10.  $W_{100\%}$  is the energy required to reach the reference density calculated by the model.

$$W \text{ (KJ/Kg)} = a \cdot C \text{ (\%)} + b \quad (6)$$

Where:

- $W$  (KJ/Kg): Compaction energy per unit mass;
- $C$  (%): Degree of compaction, calculated as the percentage of the density achieved in each cycle divided by the reference density at 170 °C (2.608 g/cm<sup>3</sup>);

- a and b are constants.

*Table 10. Required energy in function of the compaction degree*

Temperature	a	b	R <sup>2</sup>	W <sub>100%</sub>
Conventional	0.143	-11.984	0.96	2.31 KJ/Kg
Reduced	0.125	-10.564	0.95	1.94 KJ/Kg

In order to confirm the significance of the results, and as they followed a normal distribution and there was homogeneity of variances, the T-Student test was carried out. The analysis showed that the compaction energy of both PA mixes (with and without wax) did not have significant differences (p-value 0.315). Accordingly, the mixture could be laid like a conventional mixture in spite of the decrease of the production temperature, as long as the temperature of the mixture with the wax is above its melting point (130 °C – 140 °C).

#### **3.4 Dynamic analysis of the mixture at reduced temperature**

In order to analyse the influence of the wax on the dynamic performance of the asphalt mixtures, the stiffness and the fatigue resistance of the PA mixtures were determined using the four point bending test according to EN 12697-26 and EN 12697-24 respectively, in a universal hydraulic machine Zwick Z100. To determine the stiffness modulus, specimens were tested at 20 °C, a fixed strain amplitude of 50 µm/m and a frequency sweep was carried out from 0.1Hz to 30Hz. Fatigue tests were carried out at 20 °C and 30 Hz, the failure criteria was defined as the load cycle when the dynamic stiffness decreases to half of its initial value, being this initial value the stiffness after 100 load cycles. There are not minimal requirements regarding stiffness and fatigue resistance for this type of mixture in the Spanish specifications, but these properties are important for the characterisation of the asphalt mixture behaviour.

##### *Stiffness. Four point bending test (EN 12697-26. Annex B)*

The dynamic modulus (E\*) and phase angle (φ) of both mixtures are presented in Table 11.

*Table 11. Dynamic modulus test*

Frequency	Conventional temperature		Reduced temperature	
	E* ± Deviation	φ ± Deviation	E* ± Deviation	φ ± Deviation

(Hz)	(MPa)		(°)		(MPa)		(°)	
0.1	734	290	41.2	3.9	379	55	38.6	3.4
0.2	782	249	40.2	3.9	476	55	38.1	2.2
0.5	929	240	38.8	3.9	665	68	37.5	1.7
1	1088	253	37.4	3.9	851	83	36.8	1.2
2	1274	267	36.1	4.2	1091	101	35.7	1.0
5	1618	314	34.1	4.4	1512	138	33.7	0.9
8	1881	391	32.8	4.6	1774	158	32.4	0.8
10	1965	383	32.3	4.5	1906	169	31.9	0.7
20	2403	471	33.0	4.5	2384	211	29.9	0.7
30	2674	518	29.7	4.5	2796	186	29.7	1.7

A slightly lower dynamic modulus was obtained for the PA mixture with wax, especially at the lowest frequencies. However, the statistical analysis carried out indicated that there are not statistical differences between the mixtures, since the significance in the U test of Mann-Whitney was 0.164. In the case of the phase angle, despite the slight decrease, the difference between mixtures is very small, so it cannot be concluded that the mixture with the wax is more elastic.

In spite of the differences shown by the binders in the rheology test (the CRM binder with wax presented a higher  $G^*$  and a lower  $\delta$ ), the addition of wax has not significantly affected the stiffness of the mixture, probably due to the fact that, in this type of mixture, this property is mostly influenced by the high percentage of voids.

However, although the differences among mixtures are very small, if we consider that this test has been carried out at 20 °C, the results of the PA mixtures followed a similar trend that the one observed with the bitumen in Figure 2 and Figure 3. In Figure 2, at low frequencies, the differences in stiffness among the bitumen and the bitumen/wax were small. However, these differences increased at higher frequencies. Regarding the PA mixes, at low frequencies, the asphalt mixture with wax presented a smaller modulus, probably due to the high percentage of voids. Nevertheless, as frequency increases, the differences between the dynamic modulus of both mixes is reduced, being the dynamic modulus of the PA mixture with wax higher at 30 Hz.

A similar behaviour is observed for the phase angle. The greatest differences between the phase angle of the CRM bitumen and CRM bitumen/wax were found at low

frequencies, while at high frequencies, similar phase angle values were obtained (Figure 3, at 20 °C). Likewise, phase angle of the PA mixes (Table 11) presented the greatest differences at low frequencies while at the highest frequencies the differences were minimum.

The correlation coefficient between the DSR test at 20 °C and the dynamic test was calculated for a range of frequencies from 0.1 Hz to 10 Hz. According to Table 12, good correlation was obtained in all cases. However, considering the small differences in stiffness and phase angle at 20 °C, the analysis of these correlations at other temperatures is recommended.

*Table 12. Correlation between stiffness and phase angle of DSR and dynamic modulus tests*

Correlation coefficient	Samples without wax		Samples with wax	
	Stiffness $G^* - E^*$	Phase angle $\delta - \Phi$	Stiffness $G^* - E^*$	Phase angle $\delta - \Phi$
$R^2$	0.97	0.98	0.96	0.98

*Resistance to fatigue. Four point bending test (EN 12697-24. Annex D)*

Table 13 presents the initial modulus ( $S_0$ ), the strain-characteristic at  $10^6$  cycles, the fatigue laws and the coefficient correlation for the PA mixture manufactured at reduced temperature with the wax. Although there are not specific requirements for the fatigue resistance of PA mixes because they are usually employed in surface layers and under compression strengths, the fatigue performance of this mixture is good and the addition of wax do not negatively affect this parameter.

*Table 13. Results of fatigue resistance of the PA mixture with wax*

$S_0$ (MPa)	Deformation* ( $\mu\text{m/m}$ )	fatigue law	$R^2$
2329	165	$\varepsilon(\text{m/m}) = 3.947 \cdot 10^{-3} \cdot N(\text{cycles})^{-0.230}$	0.79

\* $10^6$  cycles

#### 4. Conclusions

A PA mixture with alternative aggregates and a CRM binder has been designed. Besides, a fatty acid amide wax has been added with the aim of reducing the production temperature of the PA mixture to the conventional ranges used by a 50/70 penetration grade bitumen. The influence of the wax on the bitumen and the asphalt mixture has

been analysed by determining the rheology of the bitumen samples and the mechanical behaviour of the PA mixtures.

Based on the results of this study, the following conclusions are drawn:

- The addition of wax produces an increase of the stiffness of the CRM binder, increasing also the elastic component and decreasing the thermal susceptibility. However, this increase has not been reflected in the stiffness of the asphalt mixture.
- Above the melting point of the fatty acid amide wax (130 °C / 140°C) a decrease of the viscosity of the CRM binder is observed that allows to decrease the manufacturing temperature of the asphalt mixture by 15 °C.
- The PA mixtures designed with 90.9% of alternative aggregates fulfilled the technical requirements established by the Spanish regulations for their use in the most demanding roads.
- The mixture at reduced temperature has not had significant differences with the mixture at conventional temperature in the mechanical tests, although it seems a tendency to increase the percentage of voids that could affect the indirect tensile strength of the samples in the water sensitivity test. Only the indirect strength of the dry samples of the water sensitivity tests was statically different. The results in the cantabro loss particle test were also similar.
- The stiffness and the resistance to fatigue have not turned out as properties that limit the use of the wax in the selected percentage of 3 %.
- The workability analysis has shown that there are not significant differences between the mixtures. The incorporation of the wax does not modify compaction energy, while this compaction is performed above 130 °C. Therefore, the compaction process of the mixture would be the same than a conventional mixture with 50/70 penetration grade bitumen.

## Acknowledgements

GREENROAD is a project financed by the “LIFE+” program of the European Union, with reference number LIFE 11 ENV/ES/623. This project was carried out by a consortium coordinated by COPSESA (Constructora Obras Públicas San Emeterio S.A.) and integrated by GITECO (Construction Technology Applied Research Group, University of Cantabria) and the Department of Roads Construction from the Santander City Council.

The authors wish to acknowledge and especially thank Antonio García Siller (CEPSA), Fernando Di Baggio (Unión Derivan S.A.), CODEFER S.L. and Global Steel Wire S.A. (GSW) for their collaboration.

This work was also supported by the Spanish Ministry of Economy and Competitiveness and the EDRF-FEDER through the research project BIA2012-32463.

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